

THE DETERMINATION OF IGNITED SOLIDS

The ignited solids test determines both the quantity of material that remains as a residue and the quantity of material that is lost when the total, suspended, or dissolved solids residue is ignited at 600°C for one hour. The test does not determine specific substances but only a general class of substances which have a similar response under the same test conditions.

The loss on ignition is due to oxidation of carbon and hydrogen, escape of water of hydration, volatilization of inorganic ammonium salts, decomposition of magnesium carbonate, and loss of any other materials which are volatile or decomposable below 600°C . Thus, the loss on ignition generally over estimates the organic content. The actual organic content is lower by the amount of water, inorganic salts, etc., released. In the case of clean natural waters, the loss on ignition is due almost entirely to non-carbonaceous material; therefore caution should be exercised in interpreting the results on such samples.

Ignited solids results are most useful in the characterization of sewage sludges where it is desirable to estimate the organic fraction. It is one of the major tests used in assessing the efficiency of sludge digestion.

1. Sample Handling and Preservation

Samples may be collected in glass bottles and refrigerated. Do not add preservatives, freeze, or fill to the brim if the sample is a sludge.

2. Selection of Method

Ignited solids are determined gravimetrically by igniting the total, suspended, or dissolved solids residue at $600 \pm 10^{\circ}\text{C}$ for one hour.

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THE DETERMINATION OF IGNITED SOLIDS
GRAVIMETRIC METHOD

SUMMARY

Substance determined.	The ignited solids test determines both the quantity of material that remains as a residue and the quantity that is lost when the total, suspended, or dissolved solids are ignited at $600 \pm 10^{\circ}\text{C}$ for one hour.
Interpretation of results.	Results are reported in mg/l where the volume pertains to the original aliquot of sample. The dried residue source is identified (total, suspended or dissolved) and both the loss on ignition and residual ash concentrations are reported.
Principal of method.	Analysis is accomplished gravimetrically by igniting the total, suspended, or dissolved solids residue at $600 \pm 10^{\circ}\text{C}$ for one hour.
Time required for analysis.	Two hours, provided the total, suspended or dissolved solids data and residues are available.
Range of application.	Test is primarily performed on sewage streams to estimate volatile organic content; when applied to "clean" samples, the loss on ignition is basically non-carbonaceous.
Standard deviation.	Ash - Suspended Solids: 5.2 mg/l - Dissolved Solids: 12.8 mg/l - Total Solids: 0.98%
Accuracy.	Not applicable.
Limit of detection.	Ash - Suspended Solids: $3.29S = 17 \text{ mg/l}$ - Dissolved Solids: $3.29S = 42 \text{ mg/l}$ - Total Solids: Insufficient Data
Interferences and shortcomings.	The inaccuracies associated with the suspended, dissolved or total solids procedures are combined in the ignition step.

Minimum volume of sample. 300 ml.

Preservation and sample container. Glass bottles are adequate, but must not be filled to the brim for sludges. Preservatives should not be added, but the sample should be refrigerated.

Safety considerations. Use asbestos gloves and crucible tongs when removing samples from the muffle furnace. The crucibles require about *one hour* to cool to room temperature.

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1. Introduction

The suspended, dissolved, or total solids residue is ignited at 600°C for *one hour*. Both the ash residual and loss on ignition are calculated, and reported as weight (mg) per original aliquot of sample (ml). Some samples, i.e. sludges, for total ignited solids, cannot be pipetted and are usually scooped into an evaporating dish and weighed prior to drying. The sample is then treated in the normal manner.

2. Interferences and Shortcomings

Any problems associated with the total, suspended, and dissolved solids determinations also affect the ignited solids test. Errors resulting from physical losses due to decrepitation are also a possibility.

3. Apparatus

- a) Muffle furnace capable of maintaining $600 \pm 10^{\circ}\text{C}$.
- b) Balance capable of 5-decimal point resolution.
- c) Petrie dishes - 9.3 cm I.D., pyrex glass.
- d) Desiccators.
- e) All equipment for suspended, dissolved or total solids tests is also required. (See appropriate test method.)

4. Reagents

None.

5. Procedure

THE DRIED RESIDUE FROM THE SUSPENDED, DISSOLVED OR TOTAL TEST PROCEDURE IS REQUIRED (PLEASE SEE THE DETERMINATION OF SOLIDS); THE SAMPLE ALIQUOT AND DRIED WEIGHT DATA ARE ALSO NECESSARY.

- a) Place dried residue in muffle and wait for temperature to reach $600 \pm 10^{\circ}\text{C}$; ignite for *one hour*.
(Fibre glass filter paper discs are kept in pyrex glass petrie dishes rather than racks so they can be heated to 600°C .) After firing, cool evaporating dishes and petrie dishes slightly and transfer to desiccators as soon as feasible. Cool to room temperature before weighing.
- b) Dust, level and zero balance (5-decimal place).
- c) Weigh quality control A & B dishes or filter papers - whichever is appropriate. Ensure that A plus B and A minus B values meet limit specifications.
- d) Weigh ignited samples. Analyze at least 4% of the samples in duplicate.
- e) Utilizing the appropriate data, calculate both the residual ash and weight loss due to ignition.

6. Calculation and Reporting

- a) Routine type samples, based on sample volume.

$$\text{Loss on ignition (mg/l): } \frac{(A - B)}{V} \times 10^6$$

where B = the weight of ash plus filter paper or evaporating dish after ignition

A = the weight of residue plus filter paper or dish before ignition

V (ml) = the aliquot of sample taken for the suspended, dissolved, or total solid determination.

$$\text{Ash (mg/l): } \frac{(B - C)}{V} \times 10^6$$

where C = the weight of the filter paper or
evaporating dish

B = the weight of the ash plus filter paper
or evaporating dish

V (ml) = the aliquot of sample taken for the
suspended, dissolved, or total solids
determination.

- b) For total ignited solids on sludges only, based on sample weight.

$$\text{Dried weight (mg/l): } \frac{(F - G)}{E} \times 10^6$$

where E = the weight of the wet sample* plus dish,
minus the dish tare

F = the weight of the dried sample plus dish

G = the weight of the dish

$$\text{Loss on ignition (mg/l): } \frac{(F - H)}{E} \times 10^6$$

where E = the weight of the wet sample* plus dish,
minus the dish tare

F = the weight of the dried sample plus dish

H = the weight of the ash plus dish

$$\text{Ash (mg/l): } \frac{(H - G)}{E} \times 10^6$$

where E = the weight of the wet sample* plus dish,
minus the dish tare

G = the weight of the dish

H = weight of the ash plus dish

Results are reported as follows:

<u>Range</u>	<u>Report to:</u>
0 - 100	2 significant figures
>100	3 significant figures

*ASSUME DENSITY IS 1 mg/ml.

7. Precision (Ash Residual)

<u>Residue Source</u>	<u>Ash Conc. Range (mg/l)</u>	<u># Samples</u>	<u>Standard Deviation (mg/l)</u>	<u>Relative Standard Deviation (%)</u>
Suspended Solids	0 - 100	25	5.2	-
Dissolved Solids	0 - 500	10	12.8	-
Total Solids	1000 - 65,000	15	-	0.97

8. Bibliography

- i) *Standard Methods for the Examination of Water and Wastewater*, 14th ed. p 89., 1975.
- ii) *Outline of Analytical Methods*, Ministry of the Environment, Feb. 25, 1975.

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